Office of Laboratory Licensure, Certification & Training
3443 N Central Avenue, Suite 810
Phoenix, Arizona 85012
(602) 255-3454
(602) 255-1070 FAX
Technical Support Hot-Line 1-800-592-0374

Jane Dee Hull, Governor
James R. Allen, MD, MPH, Director

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TO: Laboratory Director and QA Manager

FROM: Dr. Barbara J. Erickson, Ph.D., Bureau Chief

SUBJECT: Information Update #47

NOTE: If any problems occur with this web site, please call 1-800-952-0074 or (602) 255-3454 and ask for Technical Resources and Training. Thank You.

1. On March 27, 1998 Arizona's Office of Laboratory Licensure and Training held a round table discussion with representatives from the environmental laboratories on Method 8000B. During the meeting several issues came up which needed further clarification. We contacted the Methods Information Communication Exchange (MICE) line to help clarify these issues. Their answers (A) are given below.

   Q. Section 7.8.2, line 7 states "when employing external standard calibration, it is necessary that a calibration verification ... bracket the sample analyses." What if you are running internal standard calibration?

   A. The end of shift calibration verification is not necessary when using an internal standard, since the responses for the internal standard themselves can be examined and evaluated.

   Q. Section 7.10.4.1 states "If one result is significantly higher (e.g. >40%), ..." Is 40% meant to be the level at which the reporting criteria in 7.10.4.2 is triggered?

   A. The 40% criteria is the cutoff. The intent of this specification was that if the difference is >40%, and no chromatographic problems were found, then the laboratory would report the higher number.

   Q. The last paragraph of Section 7.7, states "If the calibration does not meet the 15% limit...check the instrument operating conditions ... restore them to the original settings" What is allowed in restoring the original settings before a new calibration curve would have to be generated?

   A. Restoring the settings means resetting the temperature program, flow rates, elution

gradient, etc., to the original settings used for the initial calibration. Some changes can occur to flow rates, etc., that will affect the calibration. This section does NOT mean that the lab can replace a GC column, clean the source, etc., without recalibration. It simply means that the analyst should recheck the settings and make reasonable adjustments to return them to "normal" without the need to recalibrate completely. There is some guidance on when the initial calibration must be repeated in Sec. 8.2.5.2. The items in that section should NOT be reset without recalibration. Sec. 8.2.5.1 provides examples (and only examples) of things that do not require recalibration automatically. Taking the instrument apart does NOT qualify, as a rule.

Q. Section 7.7.6, 3rd paragraph, line 6 states "... if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., > 15%, and the analyte was not detected in any of the previous samples during the analytical shift, then the sample extracts do not need to be reanalyzed... " Is this the only situation where the verification standard can be outside the acceptance criteria and the previous samples do not need to be reanalyzed?

A. The short answer is "no." There are probably other instances in which it would not be necessary to rerun a group of samples. Another example would be when the samples are all above the upper limit of the calibration range, thus requiring dilution before useful data can be obtained. They might be "rerun" at a dilution, but it would not make sense to rerun the original extracts without dilution just because the calibration verification standard was a bit off.

2. We've noticed that the 600 and 8000 series methods only make recommendations regarding the running of trip blanks (EPA refers to this as field reagent blanks). The Arizona Office of Laboratory Licensure has adopted the requirements found in the Technical Notes on Drinking Water Methods, October 1994. These requirements are:

   "If a water sample is contaminated with an analyte, verify that it is not a sampling error by analyzing a field reagent blank. The results of these analyses will help define contamination resulting from field sampling, storage and transportation activities. If the field reagent blank shows unacceptable contamination, the analyst should identify and eliminate the contamination."

3. The laboratories are uncertain about the steps to be taken to add a method not listed in the Licensure Rules or to add a specific analyte not listed in the reference method, to their license. A detailed narrative can be found in the Environmental Laboratory Licensure Rules, Section R9-14-608, B, Approved Methods and References. Address the petition to Steve Baker, Program Manager, Environmental Laboratory Licensing.

4. Per Jim O'Dell of USEPA, Cincinnati, there is a typographical error in the 40 CFR, Part 136.3, List of Approved Inorganic Test Procedures, Table 1B for Waste Water parameters.

   #31. Kjeldahl Nitrogen-Total (as N), mg/L: Digestion and distillation followed by:.....351.3 or SM 4500-NH3 B or C.

   The SM method should read as 4500 N_{ORG} B or C.

5. For Methods 8260B and 8270C, all parameters of interest must be included in the 12 hour calibration verification standard. Since these methods only have acceptance criteria for CCCs and SPCCs the following Arizona Environmental Laboratory Rule applies for all other compounds and will be enforced.

   Section R9-14-613, B9 states " If a laboratory tests for a parameter for which quality control acceptance criteria is not specified, the laboratory must statistically develop limits from historical data. The mean and standard deviation for a minimum of twenty points, excluding statistical outliers, must be determined. The limits shall be no more than 3 standard deviations from the mean and shall be in the detectable range".

6. We have received several questions regarding the quantitation requirements for N-Nitrosodiphenylamine by 8270C. ADHS has consulted with the MICE hotline. The CCC (Calibration Check Compounds) for N-Nitrosodiphenylamine was changed to Diphenylamine because of the degradation problem. The Office of Solid Waste expects N-Nitrosodiphenylamine to be monitored as Diphenylamine. It is not necessary to have a separate Diphenylamine standard for calibration and verification. For reporting purposes, it can either be reported as N-Nitrosodiphenylamine or as a pair of N-Nitrosodiphenylamine/Diphenylamine.

7. On the following page we are requesting information and input from the laboratories who participated in the 8015AZ Blind Study or involved in the method development. The information obtained will be utilized in the revision of Method 8015AZ. Check off all that apply. Please fax the completed survey to (602) 255-1070 ASAP.

8. If you have any questions regarding the Updates, or if you have any technical questions that need clarification, please call or send e-mail to Prabha Acharya, Program Manager, Technical Resources and Training at the Laboratory Licensure. A table of contents to all the Information Updates published is also available.